OXFORD CRYOSYSTEMS

N-HeliX

Operation & Instruction Guide

OXFORD CRYOSYSTEMS N-HELIX

Operation & Instruction Guide v1.7

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1. Introduction

Welcome to the Oxford Cryosystems N-HeliX operating and instruction guide. The N-HeliX is designed for use in the freezing of macromolecular, small molecule and powder samples during the collection of x-ray data with either helium or nitrogen.

It is capable of cooling samples to 28 K in an open stream of dry helium gas and with nitrogen gas at higher temperatures. Icing and unwanted moisture is prevented from reaching the sample by a concentric shroud of dry helium at room temperature surrounding the inner core of cold gas. Icing of the nozzle is prevented by an integrated nozzle heater, which maintains the nozzle temperature at around 310 K.

1.1. How the N-HeliX works

Figure 1 illustrates the gas flow circuit of the N-HeliX.

Figure 1 – N-HeliX layout

Bottled helium gas and/or nitrogen gas is cooled by passing it through heat exchangers mounted on a two stage, closed cycle cooler. The cold helium or nitrogen gas then passes out of the nozzle and over the sample.

The closed cycle cooler is mounted within the body of the N-HeliX and operates using compressed helium gas provided by the Cryodrive 3.0 kW compressor, which is water-cooled. The helium in this circuit is unrelated to the cold flow and is recycled by the compressor. See Figure 2 for an external view of the N-HeliX.

Figure 2 – N-HeliX external view

A Pfeiffer Turbomolecular vacuum outfit is used to continuously pump the vacuum space around the N-HeliX internals to minimise unwanted heat leaks into the system. It is important that a good vacuum ($<10^{-4}$mbar) is maintained with the Coldhead otherwise achieving extremely low temperatures at the sample becomes impossible.

The N-HeliX can deliver the helium at 28 K with a stability of 0.3 K while still keeping the helium gas consumption to a minimum. A standard large high-pressure gas cylinder will last approximately 20 hours, but, as with the Cryostream, the sample temperature is not affected by switching the supply of helium gas, thus enabling long term experiments to be performed. In addition, the system will automatically switch from nitrogen to helium when the gas temperature drops below about 95 K.

A second gas stream at room temperature provides extra shielding for the cold stream in order to improve the resistance to icing. A 10 mm diameter X-ray transparent Beryllium shield tube extends around and beyond the crystal to protect the cold stream from atmospheric contamination. Notches within the tube walls prevent primary X-ray beam diffraction and also allow accurate crystal positioning.

1.2. Items required for assembling your N-HeliX

The component parts of the system are:

- The N-HeliX Coldhead
- 700 Series N-HeliX Controller
- Interconnecting tube set:
  - One long stainless steel 6 mm tube (supply → controller)
  - One long PTFE 6 mm tube (supply → controller)
  - One short 6 mm tube (core stream delivery)
  - One 6 mm tube with 4 mm connector (shield stream delivery)
- Controller to Cryodrive serial cable
- Cryodrive to Coldhead cable
- Controller to Coldhead cable
- Oxford Cryosystems Cryodrive 3.0 kW compressor
- Stainless steel flexible helium lines (x2)
- Manual pack

Optional extras:

- N-HeliX support stand
- Turbomolecular vacuum outfit and vacuum line

2. Getting Started

2.1. Matching the N-HeliX Coldhead and 700 Series N-HeliX Controller

The N-HeliX Coldhead and 700 Series N-HeliX Controller are supplied as a matched pair - the individual characteristics of the Coldhead sensors are programmed into the Controller. Please contact your supplier if you believe you may have unmatched units.

2.2. Operating voltage

It is essential that the N-HeliX is configured to operate on the local mains electrical supply.

Item	Operating voltage requirements
N-HeliX Controller	The voltage selector switch on the rear panel must be set to the correct position, 200-240 Volts AC, 50 Hz, 3 Amps 100-120 Volts AC, 50-60 Hz, 6 Amps. Make sure an 'Anti-Surge' (T) type fuse of the correct rating is fitted.
Cryodrive 3.0kW	User configurable Supply voltage: 200, 220 or 240 V at 50 Hz or 200, 208 or 220 V at 60 Hz Supply voltage tolerance: +10 % Maximum supply fuse rating: 30 A Recommended cryocontroller fuse type: Slowblow Maximum cryocontroller fuse rating: 5 A Over voltage category (IEC664): 2
Oxford Cryosystems/ Pfeiffer Vacuum System	90-260 V 50/60 Hz. Ensure that the Diaphragm Pump on the Vacuum System is set to the correct voltage. This can be done by setting 115 or 230 on the diaphragm pump. See *Oxford Cryosystems/ Pfeiffer Vacuum Trolley* below.

Oxford Cryosystems ships all Cryodrives set to 240 V and 50 Hz as this is how they are commissioned in the UK. It will be necessary to refer to Section 2.3 to set the Primary Tap Connections for the local voltage and frequency.

2.3. Electrical supply connection for Cryodrive

We recommend that you use a suitably fused isolator at your electrical supply outlet. Locate the isolator switch close to the electrical outlet. It is also recommended that back-up fuses are installed at the electrical supply outlet. You must configure the Cryodrive to suit your electrical supply. The Cryodrive is despatched configured for use with 240 V and 50 Hz electrical supply.

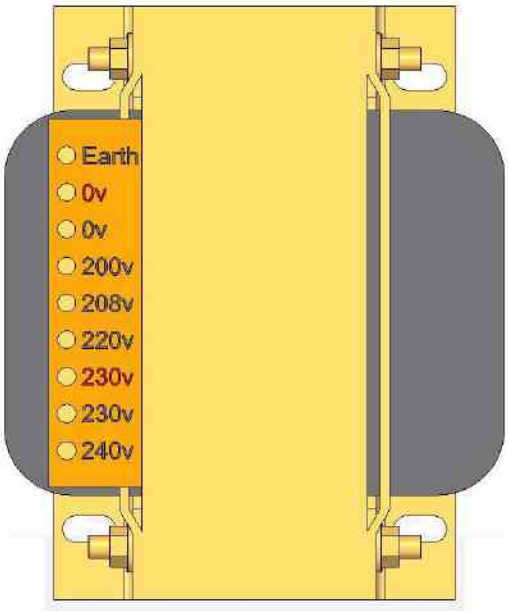


Figure 3 – Transformer showing different electrical supply connections

Use the procedure below to change this configuration.

- Look at Table 1: find your electrical supply in the left-hand column, look along this row to find the connections you must make for the wire 30 (W30) and wire 31 (W31).

- Remove the lid of the Cryodrive and locate the transformer.

- With reference to Figure 3, change the position of wire 30 (W30) and wire 31 (W31) so that they are in the correct position for your electrical supply. Ensure the connections are fully tightened. **Do not move the wires attached to the red 0 V and 230 V connectors, as these provide power to the secondary stepper motor transformer and are not part of the procedure.**

- Locate the protection switch on the current-limit potentiometer which is situated in the CryoController, refer to Figure 4. The switch can be accessed by removing the lid of the CryoController unit.

- Look at Table 2: find your Cryodrive type and electrical supply frequency in the left-hand column, look along this row to find the recommended limit for the protection switch current.

- Use a small screwdriver to adjust the current-limit potentiometer to the recommended value.

- Replace the lid of CryoController and the Cryodrive.

- Make sure that the Cryodrive ON/OFF switch is in the OFF position and connect the Cryodrive to your electrical supply.

Electrical Supply	Primary Tap Connection					
	N	200 V	208 V	220 V	230 V	240 V
50 Hz, 200 V	W32	W31	W30	-	-	-
50 Hz, 220 V	W32	-	W30	W31	-	-
50 Hz, 240 V	W32	-	W30	-	-	W31
60 Hz, 200 V	W32	W31	-	-	W30	-
60 Hz, 208 V	W32	-	W31	-	W30	-
60 Hz, 220 V	W32	-	-	W31	W30	-
60 Hz, 240 V	W32				W30	W31

Table 1 – Wire connections to primary tap connections

Cryodrive model	Electrical supply voltage			
	200 V	208 V	220 V	240 V
Cryodrive 1.5, 50 Hz supply	11 A	-	10 A	10 A
Cryodrive 1.5, 60 Hz supply	11 A	11 A	11 A	-
Cryodrive 3.0, 50 Hz supply	16 A	-	16 A	16 A
Cryodrive 3.0, 60 Hz supply	18 A	17 A	16 A	-

Table 2 – Recommended protection switch current limit setting

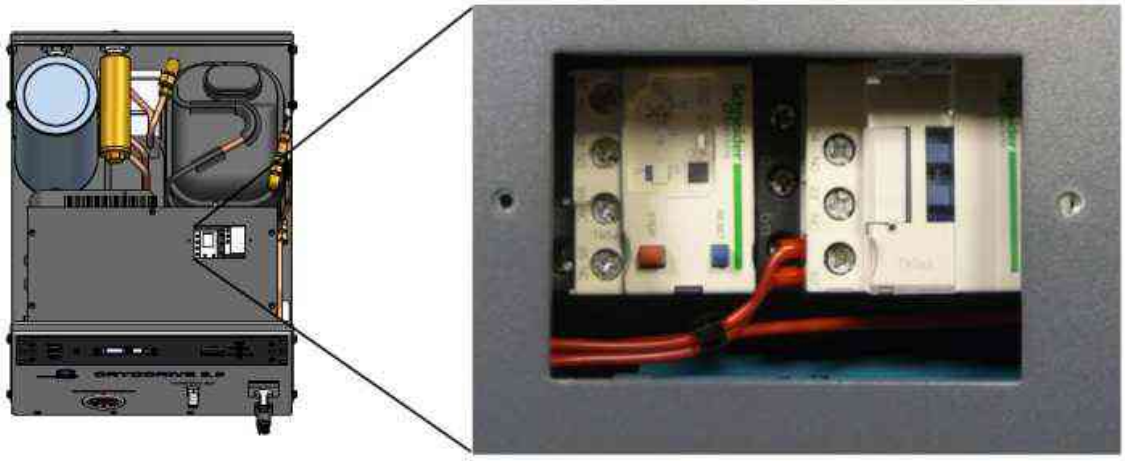


Figure 4 – Overload relay showing stop and reset button

3. Setting Up

3.1. Mounting of the N-HeliX Coldhead on the X-ray System

The mounting of the N-HeliX Coldhead depends on the particular x-ray system being used.

NOTE

The following rules and instructions are guidelines only and if a user has an alternative technique for mounting the n-HeliX then they are welcome to use it. If the user is in anyway unsure of the mounting of the n-HeliX they should contact their local n-HeliX supplier or Oxford Cryosystems for advice.

3.1.1. General rules

There are a few general rules the user should consider when mounting the Coldhead and fixing the support stand in position.

1. Do not point the helium cold stream directly at the detector.
2. Try to limit the amount the N-HeliX nozzle infringes the path of the x-rays.
3. Do not point the cold stream at any optical device or gearing (these devices need to be more than 15 cm away).
4. Do not mount the cold stream coaxial with the goniometer head as this may cause icing problems.
5. Do not fix the stand to your cabinet top so it prevents access to your x-ray tube, prevents the detector being swung in theta or makes access to the crystal difficult.
6. The N-HeliX Coldhead should only be mounted vertically to achieve correct stream projection.
7. The crystal should be mounted to the vertical centre line of the N-HeliX, and at the optimum position as shown below in Figure 5.
8. The beryllium notches can be adjusted by rotating the cone to which it is attached. They should be aligned to the incident X-ray beam.

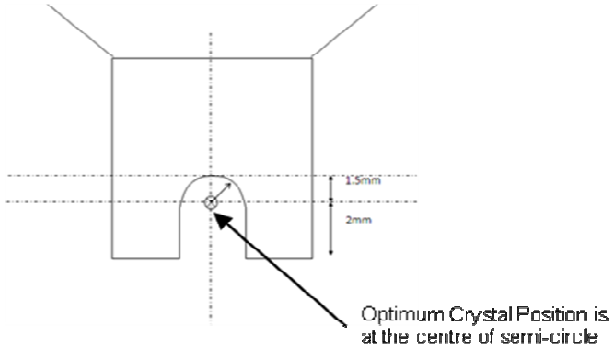


Figure 5 – Optimum crystal position

3.2. Connecting up the N-HeliX

3.2.1. Order of assembly

The main layout is illustrated schematically in Figure 1. Connections are generally labelled equivalently at each end. Start with the two high-pressure hoses between the coldhead and the compressor module. The ends are colour coded to avoid confusion: the helium return line has a GREEN band, helium supply line has a RED band, see Figure 6. When tightening the nut hold the hose in alignment with the fixed coupling to prevent loss of helium gas and do not allow the hose fitting to rotate. More information on the high-pressure hoses is detailed in the accompanying Cryodrive manual.

Figure 6 – High pressure delivery hoses

Next, fit the gas delivery tubes. The helium exchange gas must be of high purity (typically 99.995%) and the cylinder should be fitted with a pressure regulator valve. Both the helium and nitrogen supplies must be set to 1 bar gauge output.

3.2.2. Cooling water connection

1. Use hose clips to secure suitable water hoses (1/2-inch nominal internal diameter) to the water connection nozzles.

2. Connect the supply and return hoses to the cooling water inlet and outlet connectors as marked on the rear of the Cryodrive.

3. Connect the water supply hose to cooling water supply with an adequate flow rate and temperature (see section 3.2.3).

4. Connect the water return hose to a suitable drain.

5. Turn on the cooling water supply and check that there are no water leaks.

3.2.3. Water cooling requirements

- Water cooling/chiller requirement: 3.0 kW
- Minimum flow rate: 1.5 L/min
- Maximum flow rate: 7.0 L/min
- Maximum water supply pressure: 101.5 psig.
- Minimum water supply temperature (at start –up): +4ºC.
- Maximum water discharge temperature: +33ºC.
- Water quality pH range: 6.0 to 8.0.
- Maximum calcium carbonate concentration: 75 ppm

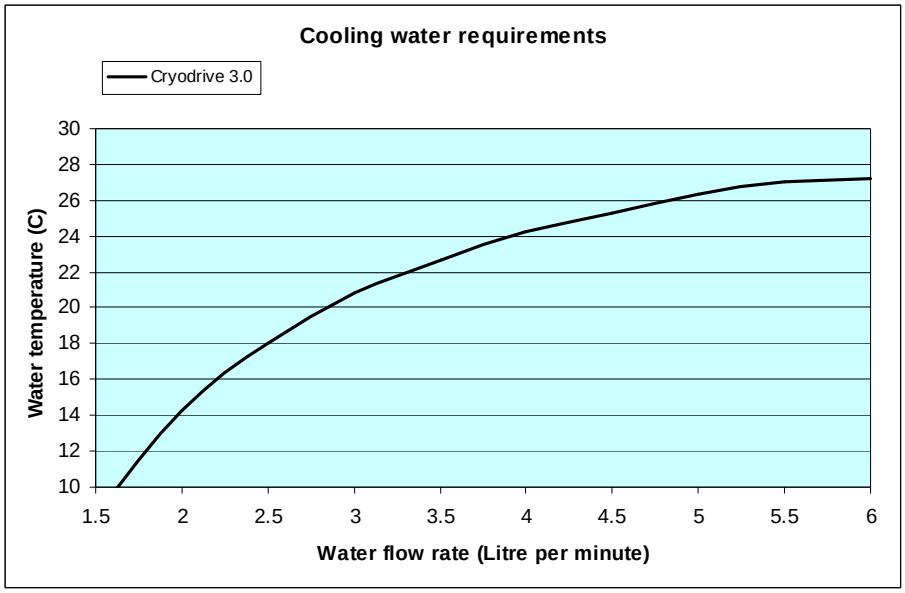


Figure 7 – Cooling water requirements

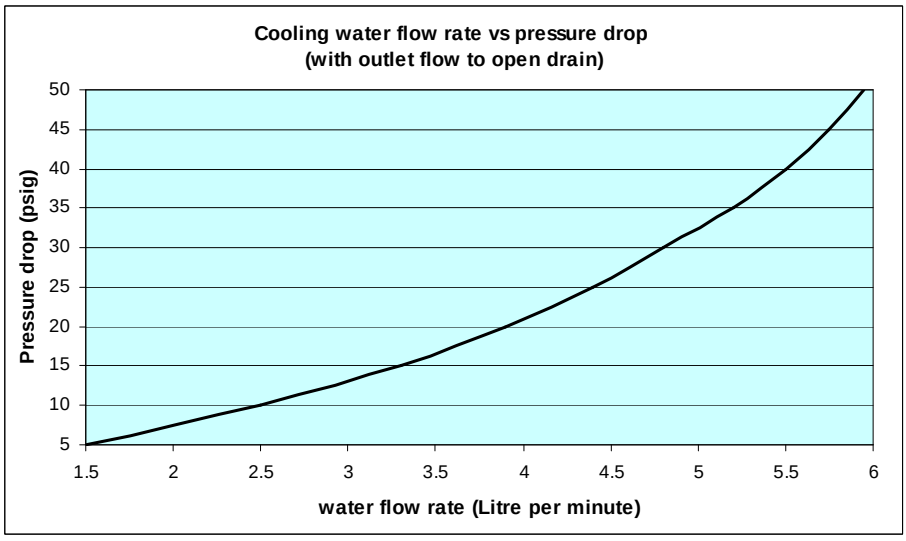


Figure 8 – Cooling water flow rate vs pressure drop

Use the following convention for fitting each tube into its connector:

Tube Type	Connector / Location		Connector / Location
Long 6 mm Tube	Helium Supply	connected to	HELIUM IN
Long 6 mm Tube	Nitrogen Supply	connected to	NITROGEN IN
Short 6 mm Tube	COLD OUT (6 mm)	connected to	N-HELIX COLDHEAD (6 mm)
Short 6 mm Tube with 4 mm connector	SHIELD OUT (4 mm)	connected to	N-HELIX COLDHEAD (4 mm)

Now fit the flexible stainless steel vacuum line between the N-HeliX Coldhead and the Turbomolecular pumping outfit. Make sure that the 'O' ring seals and flanges are very clean before assembly. The pumping outfit must be assembled as shown below before use. Note the position of the Penning gauge and reducer fittings.

Connect the signal cables and the mains power cables. The COLDHEAD 1 and PCSP sockets are on the front of the Cryodrive enclosure, see Figure 9.

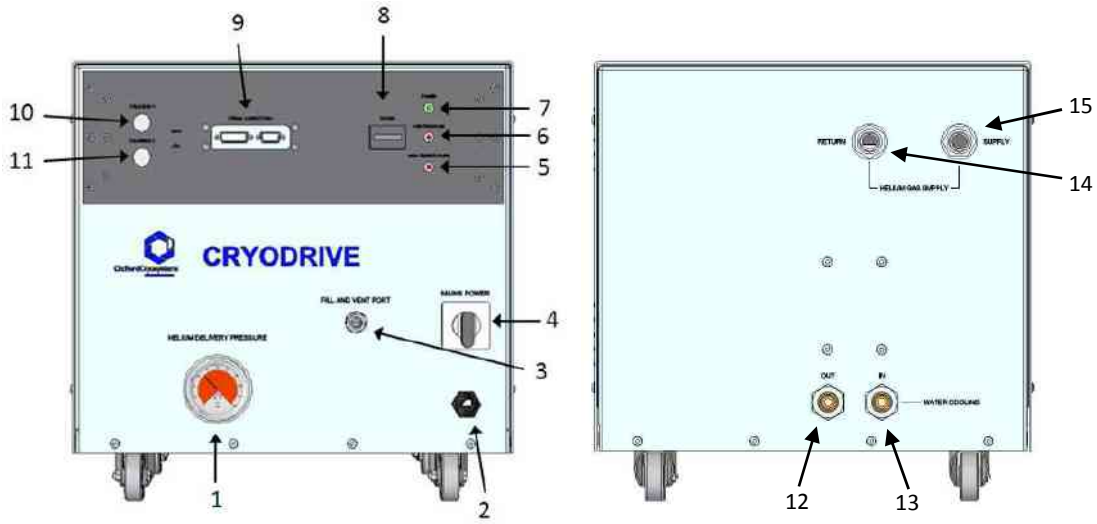


Figure 9 – Front and back of Cryodrive 3.0

1. Pressure gauge
2. Mains supply in
3. Charge and vent port
4. ON/OFF (reset) switch
5. High temperature LED
6. Low pressure LED
7. Cryodrive ON LED
8. Elapsed hour counter
9. Serial cable connections
10. Coldhead 1 connection
11. Coldhead 2 connection
12. Cooling water outlet
13. Cooling water inlet
14. Helium return
15. Helium supply

IMPORTANT: If the Cryodrive is used for the first time

It is important to check the charge pressure of the Cryodrive compressor. In the quiescent state, the pressure should read approximately 16.5 bar ± 1.0 bar. If this pressure reads less than 15.5 bar, contact Oxford Cryosystems. This check should be repeated, if possible, each time unit is to be turned on.

Whilst the Cryodrive is running, the pressure indicated is significantly higher than the quiescent value of approximately 16.5 bar. Under normal operating conditions it should be about 22 bar and will noticeably oscillate by around 0.5 bar. It is good practice to monitor the Cryodrive charge pressure, especially if the system has been dismantled and reconnected recently, as gas can be lost during the attachment and disconnection of the high-pressure hoses. As mentioned before – if the pressure is observed to have fallen, contact Oxford Cryosystems to arrange recharge.

3.3. Vacuum system of the N-HeliX

Oxford Cryosystems currently provide the Pfeiffer turbomolecular pumping station shown in Figure 10.

Figure 10 – Pfeiffer turbomolecular vacuum pump

NOTE

A variety of manuals are supplied with the Vacuum System for its components. The information below outlines the simple steps needed to use this system with the N-HeliX. Further information is available in the manuals, if necessary.

The Pfeiffer vacuum system is designed as a user-friendly system, which is very easy to operate. The unit comprises a Turbo Station with integrated Diaphragm Pump along with a wide range gauge and a Display and Operating Unit.

Both of these pumps are independent of one another. Although the mains power is fed through a common junction box, both the Cryo Vac Turbo Station and Seal Vac Diaphragm Pump have separate power switches as indicated in Figure 11 below.

Figure 11 – Power Switch for Turbo Station and integrated Diaphragm Pump

Unpack the Vacuum System and ensure that all packing material has been removed. Fit the Wide Range Gauge head and 'T' assembly to the top of the Turbo Station and using the supplied vacuum hose and fittings, connect the vacuum line from the Turbo Station to the N-HeliX. This is normally performed by the Installation Engineer.

The Display Unit on the front of the Turbo Station then goes through an initialisation process. Once the system has completed its initialisation process, the screen should say:

001: Heating

off

340: XXXX mbar (where XXXX is a pressure reading)

3.3.1. Turbo station display unit

1. LCD Display
2. Status Display
3. "Error Acknowledgement" key
4. Key "Left"
5. Key "Right"
6. "Turbo Station Start/Stop" key
7. Red illuminating diode for error status
8. Green illuminating diode for operating status

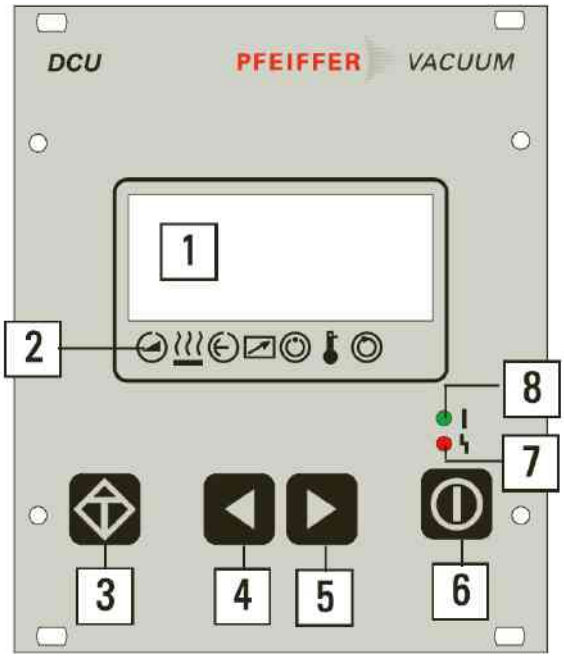


Figure 12 – Turbo station display unit

3.3.2. LED status

The red LED (error status) and green LED (operational status) can take on the following conditions:

Red LED

Illuminates: Collective error messages
Flashes: Warning

Green LED

Illuminates: Mains power unit OK, pumping station ON
Flashes: Mains power unit OK, pumping station OFF
Blinks: Mains power failure

3.3.3. Symbols

	Symbol	Arrow	Explanation
	Pump acceleration	No arrow	Not accelerating
			Accelerating (flashing)
	Heating	Not used for N-HeliX	
	Standby	No arrow	Not in standby mode
			In standby mode
	Under remote control	Not used for N-HeliX	
	Switch-point attained (represents	No arrow	Switch-point not attained
			Switch-point attained
	Excessive temperature	No arrow	Temperature OK
			Excessive pump temperature
			Excessive temperature of pump electronics
			Excessive temperature of pump and pump electronics

	Final rotation speed attained	No arrow	Final rotation speed not reached
			Final rotation speed reached

By pressing the Left or Right arrow keys, it's possible to scroll through a series of menu options.

NOTE

The Turbo Station from Pfeiffer is highly configurable and there are many options available to a user of this system. However, the system has been preconfigured by Oxford Cryosystems so there should be no need to adjust the settings of the system.

Scroll through to select '**309: Act rotspd**' which will indicate the rotation speed of the turbo when being used. It is recommended that the system is left on this menu item when running.

Press the 'Start' – 'Stop' key on the Turbo Station. This will start the Turbo Station.

3.3.4. Ballast valve

The pumping of air can cause moisture/vapour to condense in the pump and can have an effect on the attainable final pressure. To expel these condensates, gas ballast air can be admitted to help obtain these final pressures. To ensure a good vacuum, open the Gas Ballast Valve for ten minutes to ensure any residual water vapour and air in the line and N-HeliX are removed. Figure 13 below illustrates how sleeve '43' should be moved to position 'Y' to open the Gas Ballast Valve. Be sure not to leave the valve open permanently. **Note: Always remember to close the ballast valve.**

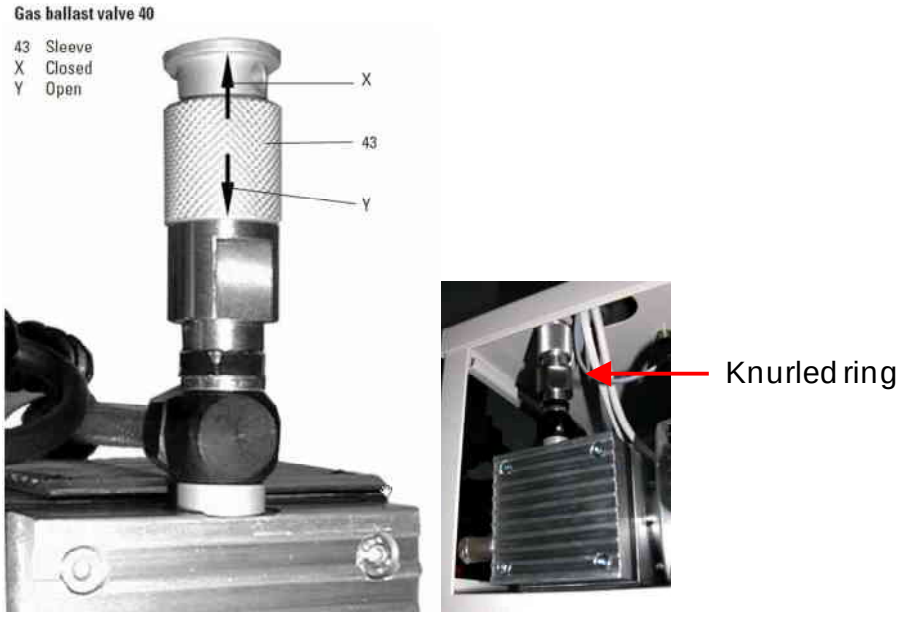


Figure 13 – Ballast valve in the closed position. To open, pull the knurled ring down by twisting it. Push or twist up to close it.

The rotation speed of the turbo pump will reach 1500 Hz. Once the pump pressure is in the 10^{-4} -10^{-5} mbar range, the N-HeliX is ready to be used.

3.3.5. Switching off the vacuum system

The system can be stopped by pressing the Turbo Station "Start/Stop" key on the Display Unit.

3.4. Check list before start up

Check to make sure that both helium and nitrogen are connected to the back of the controller and that both gas valves from the supply are open. If a nitrogen generator is being used, make sure it is switched on.

WARNING

If the system is only being use with helium it will be necessary to connect helium to both the NITROGEN IN and the HELIUM IN using some kind of splitter. The system will not work if there is only helium fed into the HELIUM IN port.

NOTE

It is very important that the helium used for the exchange gas flow is of high purity otherwise the gas flow path may block with frozen impurity. The level of purity required will depend on the duration of your low temperature measurement – most special gas suppliers offer 99.995% grade helium and this should be tried initially.

Ensure the Cryodrive compressor has adequate cooling water (check the Cryodrive manual for more information). Press the 'ON' switch for the N-HeliX Controller, which is on the right hand side of the rear panel of the Controller.

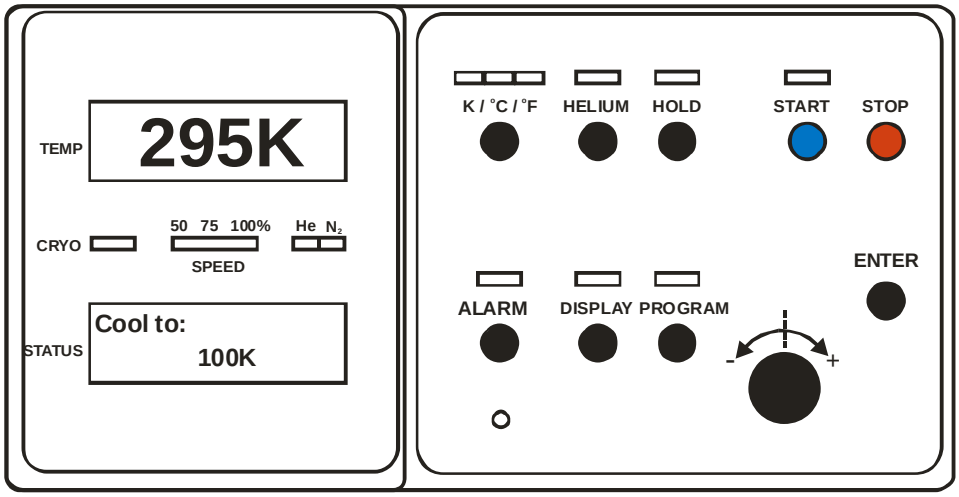
Now switch the Cryodrive on using the large switch on the right hand side of the Cryodrive. If the unit has been connected up correctly, the Cryodrive should initialise, but not start running. This is because the action of it being plugged into the N-HeliX Controller holds the Cryodrive off.

Figure 14 – N-HeliX Controller

The N-HeliX Controller is designed to provide a completely flexible means of controlling temperature. This is achieved by allowing the user to enter multiple phases.

3.5. N-HeliX controller initialisation and screen options

As the Controller is switched on, it undergoes an Initialisation process and a Self Check procedure. During the Self Check, the Controller checks to make sure all parts of the N-HeliX system are working properly.



- TEMP screen. Whilst the N-HeliX is running this screen displays the temperature of the gas stream. During start-up and shutdown the screen is used for status messages.

- CRYO/SPEED/GAS meter. The CRYO lamp indicates that the Cryodrive is working. The SPEED shows how fast the Cryodrive is operating. The GAS lamps indicated which gas is flowing through the system. It detects nitrogen and helium automatically. The lamps do not reflect the choice/selection of gas only what the system detects. When gases change over it is possible to see both lamps on. Also see *HELIUM button* below.

- STATUS screen. This screen displays information described in detail under the heading *Display Modes*.

- K/ºC/ºF button and lamps. The button allows the temperature units used by N-HeliX to be switched between Kelvin, Centigrade and Fahrenheit **at any time**. The current choice of unit is indicated by the illuminated K/ºC/ºF lamp. For the purposes of this manual, temperatures are indicated in Kelvin (K).

- HELIUM button and lamp has three states: OFF – indicates the gas choice is automatic based on temperature and is choosing to use nitrogen; FLASHING – indicates the gas choice is automatic based on temperature and is insisting on the use of helium as the system is too cold for nitrogen; ON – the user has insisted on the use of helium at all temperatures. Note: You cannot insist on nitrogen at all temperatures.

- HOLD button and lamp. Pressing the HOLD button will execute a Hold (see *Programming the N-HeliX*) and illuminate the HOLD lamp. If N-HeliX is already in a Hold, pressing HOLD again will release it.

- START button and lamp. The START button switches the N-HeliX on, executing the start-up phase or the current Phase Table (See *Programming the N-HeliX*). This button is also used to re-start the control program after it has been halted.

- STOP button. The STOP button will immediately halt the N-HeliX, turning off the Cryodrive and all the heaters. The Controller may then be safely switched off, or else re-started by pressing START.

NOTE

The approved method of shut down is via an END phase

- ALARM button and lamp. If an alarm condition develops (see *Alarm Conditions, Section 4.6*), the ALARM will be illuminated and a buzzer may sound. Pressing the ALARM button will display the cause of the alarm in the STATUS screen, and will also cancel the buzzer.

- DISPLAY button and lamp. The DISPLAY button is used to toggle the Display Mode (see *Display Modes*), indicated by the corresponding lamp.

- PROGRAM button and lamp. The PROGRAM button is used to toggle Program Mode (see *Programming the N-HeliX, Section 4*), indicated by the corresponding lamp.

- IntelliKnob. This knob is used to scroll the contents of the STATUS screen. In Program Mode it is also used together with the ENTER button to input information. The IntelliKnob is speed sensitive. This means the faster the Knob is turned, the greater the increment in the number and the slower the Knob is turned, the smaller the increment in the number.

- ENTER button. This button is used during Program Mode (see *Programming the N-HeliX, Section 4*) to input information.

3.6. Display modes

The STATUS screen displays a variety of information depending on the Display Mode and whether N-HeliX is **Running** or **Idle**. In each case the contents of the STATUS screen may be scrolled using the IntelliKnob.

The various situations are summarised below.

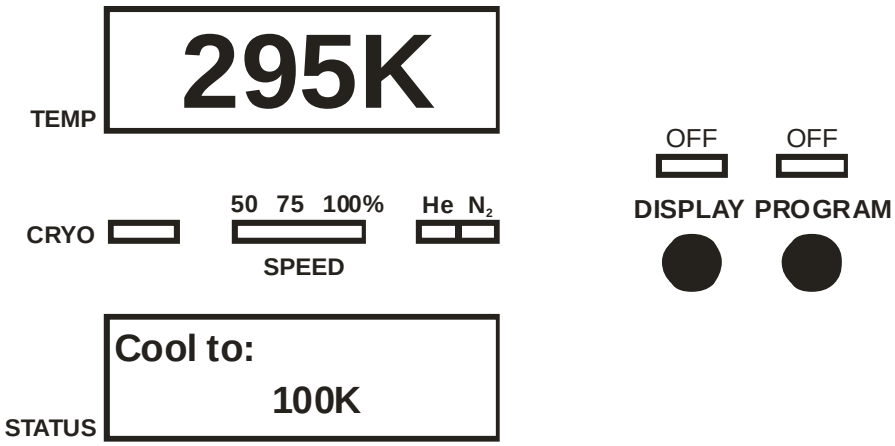
3.6.1. Display Mode 1

N-HeliX State: **IDLE (Power on, not running)**

DISPLAY Lamp: **OFF**

PROGRAM Lamp: **OFF**

Description:



Idle Phase Table Mode
If a program has not been entered, use the IntelliKnob to adjust the temperature and press START to begin. The N-HeliX will then enter a COOL function and achieve the required temperature as quickly as possible. The system automatically selects which type of gas to use.

If a program has been entered, use the IntelliKnob to scroll the STATUS screen and press START to begin.

Press DISPLAY or PROGRAM to enter the modes below.

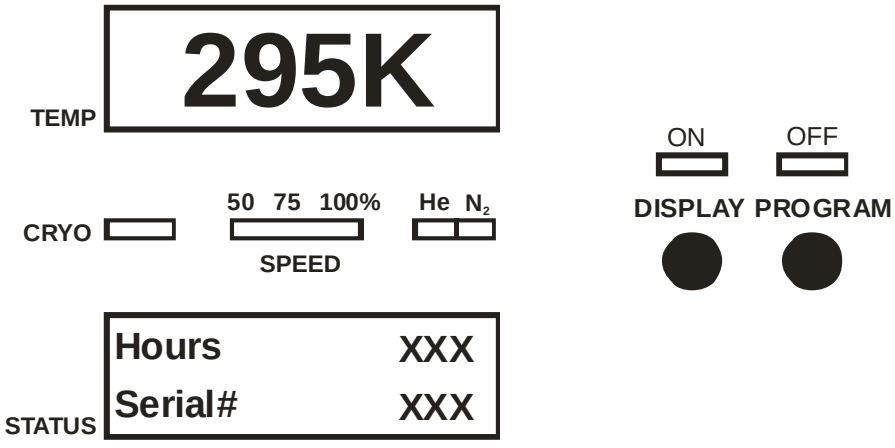
3.6.2. Display Mode 2

N-HeliX State: **IDLE**

DISPLAY Lamp: **ON**

PROGRAM Lamp: **OFF**

Description:



Idle Phase Table
Use the IntelliKnob to scroll through the following information:

Hours – the cumulative time the system has run since manufacture.
Serial# - Controller Serial Number
Software – the version of the Controller software
Shutdown – This indicates the last reason for shutdown. Options are:

STOP – The STOP button has been pressed
END – The system has been shut down due to a programmed END
PURGE – The system has been shut down due to a programmed PURGE (see *Further Programming on the N-HeliX Controller*)
POWER – The power has been switched off at the mains
FLOW – Shutdown due to low flow
TEMP – There has been a large temperature error
SENSOR – This indicates ADC latches or a sensor failure
SINK – Controller Overheating
PSU – Power Supply Overheating

LAST ERR – this stores the last reason for the shutdown but does not include STOP or POWER.

If there has been an unexpected shutdown, the following list of items is recorded on this list after the LAST ERR:

Set T, Gas T, Cryo Speed, Cryo Status, Shield T, Shield Heat, Cryo Shift, Gas Type, Gas Flow, Pressure, Outer Flow, Nozzle T, Nozzle Heat, Run Time.

These are recorded to allow the user to diagnose the reason for the shutdown and are stored until there is another erroneous shutdown.

Press DISPLAY or PROGRAM to alter the Display Mode.

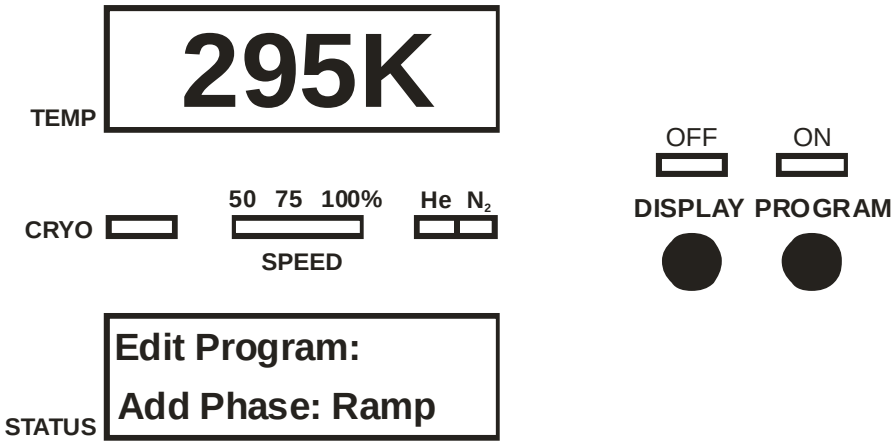
3.6.3. Display Mode 3

N-HeliX State: **IDLE**

DISPLAY Lamp: **OFF**

PROGRAM Lamp: **ON**

Description:



Program Mode

This mode allows the user to program the N-HeliX as described in *Programming the N-HeliX (Section 4)*. The list of phases also gives you the option to save or load a program. 'Save Program' will save the current program and 'Load Program' will load the last saved program.

Press DISPLAY or PROGRAM to alter the Display Mode.

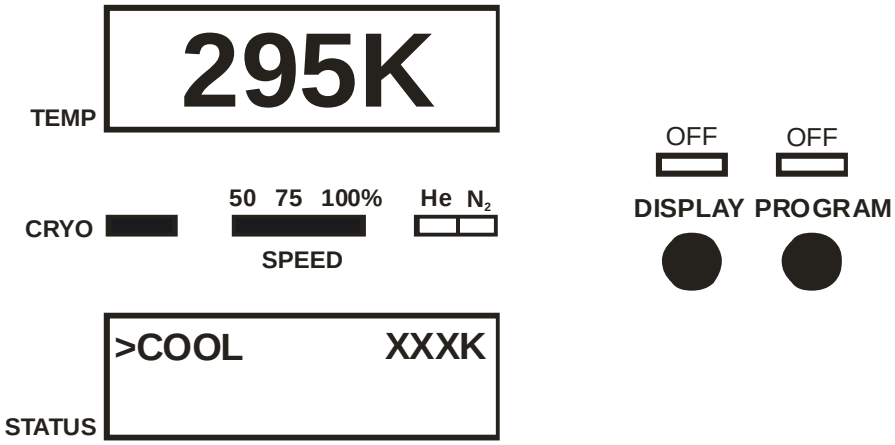
3.6.4. Display Mode 4

N-HeliX State: **RUNNING**

DISPLAY Lamp: **OFF**

PROGRAM Lamp: **OFF**

Description:



Phase Table Mode

The N-HeliX lists the phases in the current program, with the current phase at the top of the list. The current phase is indicated with a '>'. Use the IntelliKnob to scroll through the list.

Press DISPLAY or PROGRAM to enter the Display Modes 5 & 6.

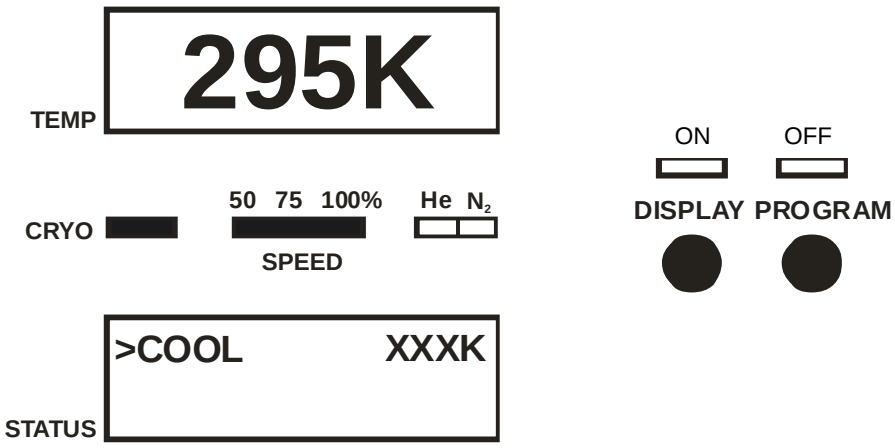
3.6.5. Display Mode 5

N-HeliX State: **RUNNING**

DISPLAY Lamp: **ON**

PROGRAM Lamp: **OFF**

Description:



Running Display Mode

Set Temp - Temperature
Temp Error - Temperature
Run Time – How long system has been running since START was last pressed.
Gas Type - Detected gas type (He/N2) as displayed via the gas type lamps

Flow Rate – gas flow in l / min
Pressure - in bar.
Outer Flow - outer gas flow in l / min
Gas Heat – %. The instantaneous value is given and the average is in brackets.
Shield T - Temperature of Cryo Shield
brackets.
Shield H - %. Current Cryo Shield heater power
Nozzle H - %. Nozzle heater power (1 or 0). Average value is indicated in brackets.
Nozzle S - Nozzle set point
Nozzle T - Nozzle temperature
Cryo - Cryodrive status
Cryo Speed - Cryodrive speed as a percentage of its maximum.
Cryo Shift - The amount by which the Cryodrive speed has been increased from its normal value in order to achieve rapid cooling. Press DISPLAY or PROGRAM to alter the Display Mode.

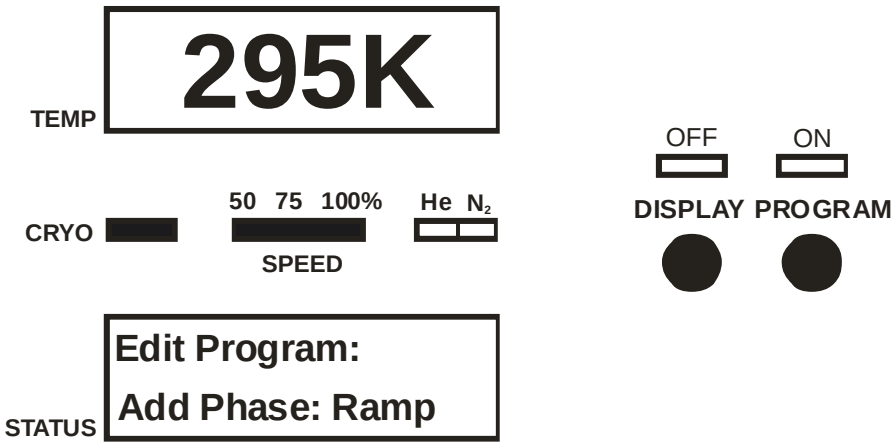
3.6.6. Display Mode 6

N-HeliX State: **RUNNING**

DISPLAY Lamp: **OFF**

PROGRAM Lamp: **ON**

Description:



Program Mode

This mode allows the user to program the N-HeliX as described in *Programming the N-HeliX*. New phases are added at the end of the list of phases. If the system is in a HOLD, press HOLD to begin the next phase.

Press DISPLAY or PROGRAM to alter the Display Mode.

4. Programming the N-HeliX

Switch the N-HeliX Controller on according to the instructions above and wait for the system to initialise.

4.1. Using the quick start facility and the COOL function

To cool as quickly as possible simply rotate the IntelliKnob to the appropriate temperature and press START. The N-HeliX will remember the last value requested here and store it for the next time the Quick Start facility is used.

NOTE

The system uses the COOL function to get cold as quickly as possible. The COOL function runs the Cryodrive at high speed to cool the coldhead as fast as possible.

Once the N-HeliX reaches the desired temperature, the Controller will automatically enter a HOLD in the Phase Table.

With the system now running, press DISPLAY to view *Display Mode 5* discussed above. This displays all the parameters of the system. Alternatively, press PROGRAM to enter *Display Mode 6* and add additional phases to your program (see *List of Phases* and *Further Programming of the N-HeliX Controller*).

4.2. Further programming of the N-HeliX Controller

While the system is idle or running, it is possible to program more detailed phases.

Press PROGRAM (this illuminates the PROGRAM lamp). The STATUS screen then displays the following:

Edit Program:
Add Phase: Ramp

Spin the IntelliKnob to see all the phases. Press ENTER at any time to accept a particular phase.

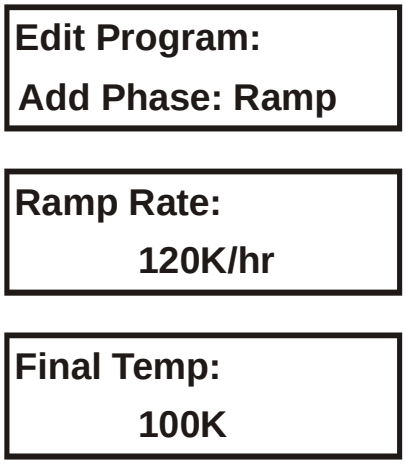
Here is a list of the possible phases and other parameters that each one requires. For an explanation of each phase, see *List of Phases*:

Phase and Description	STATUS Screen Modes

Phase: RAMP

Description
Change temperature at a controlled rate. When ramping down in temperature, if the selected rate is too fast for the N-HeliX Cooler to follow, the Controller will automatically enter the RAMP/WAIT mode (this will be indicated on the screen). The effect of this is to stop the ramp in order for the gas temperature to catch up to within 5 K of the gas temperature.

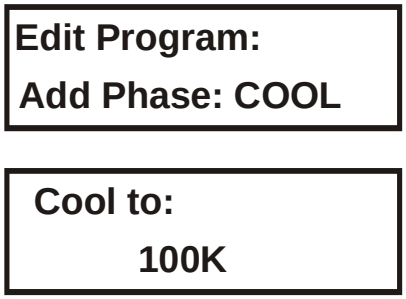
The Ramp Rate may be anything between 1 and 360 K per hour.



Phase: COOL

Description
COOL is designed to get the system as cold as quickly as possible.

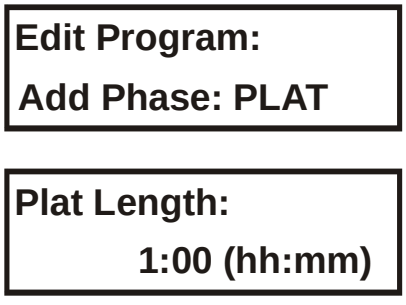
It is not possible to spin the IntelliKnob above the end temperature of the previous phase or the current gas temperature.



Phase: PLAT

Description
Maintain temperature fixed for a certain time. The user is prompted to enter a temperature at which to plateau and to specify a time to plateau.

Below 10 hr 00 min the PLAT function will start to count down in seconds and this will be displayed in the STATUS screen during running.



Phase: HOLD

Description
Maintain temperature fixed indefinitely until the START button is pressed (a programmed HOLD should not be confused with the HOLD button).

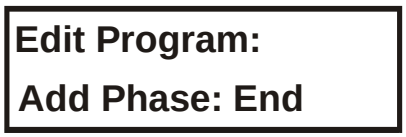
Phase: PURGE

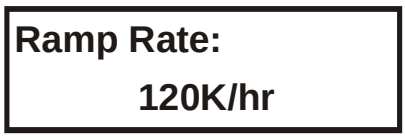
Description
This function is designed to warm up the Coldhead as quickly as possible. It applies maximum power to the heaters in the Coldhead to get to 300 K as quickly as possible. The PURGE is replaced by a SOAKING as the heaters run for a further 10 minutes.

Phase: END

Description
System shutdown. You are asked to enter a Ramp Rate back to a final temperature of 300 K and then the system is shut down. This is the controlled way to finish an experiment and should be used whenever possible. The flow rate of the N-HeliX is reduced to 1 L/min.

Once an END function has been programmed, it is not possible to enter any more phases. The only options available are to load or save a program or delete the last phase.



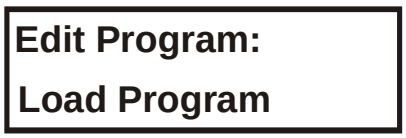


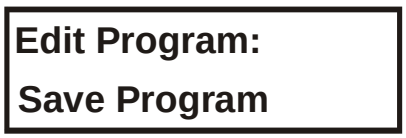
Delete Phase

Description
To delete a phase at any time, in Program Mode, spin the IntelliKnob to 'Delete Phase' and press ENTER. This will delete the last phase entered. If the system happens to be executing this last phase, the phase will be replaced by a HOLD.

Load Program / Save Program

Description
While the system is idle, it is possible to load or save a program. Loading a Program simply loads the last saved program.





NOTE

Turning the IntelliKnob also offers the option to load a program or save the current program. This is only possible when the system is idle.

Press ENTER on completing each screen. To cancel programming at any time, press PROGRAM or Display, (the PROGRAM lamp will go out).

Once the phases have been entered, press START to begin the first phase in the Phase Table. The Controller will automatically enter a HOLD phase at the end of the program if one has not already been programmed. If the system is running and already in a HOLD phase, pressing the HOLD button will pass to the next instruction in the Phase Table.

4.3. Phase table

Press PROGRAM at any time during running to look at the Phase Table and enter more phases. This will enter *Display Mode 6* but will not give the option of loading or saving a program while the system is running.

If there is a list of phases longer than the screen in the Phase Table, this will be indicated by small characters on the left of the STATUS screen pointing up or down.

↑ ↓	This indicates it is possible to scroll up and down through the phases using the IntelliKnob.
↓	This indicates it is possible to only scroll down through the phases using the IntelliKnob.
↑ ⊥	This indicates that the end of the Phase Table has been reached and it is only possible to scroll up through the phases using the IntelliKnob.
⊤	This indicates that the top of the Phase Table has been reached.
>	This indicates the current programme running.

4.4. Using Helium or Nitrogen and the Helium button

N-HeliX can operate with a choice of two gases; either helium or nitrogen. The system will only allow you to use a gas that is appropriate at any temperature. This means, the N-HeliX will avoid a situation where nitrogen is used at a temperature that will condense the nitrogen to prevent the system blocking. There are three states of the HELIUM button lamp:

OFF – indicates the gas choice is automatic based on temperature (in the region of ~90 K) and is choosing to use nitrogen.

FLASHING – indicates the gas choice is automatic based on temperature and is insisting on the use of helium as the system is too cold for nitrogen.

ON – the user has insisted on the use of helium at all temperatures. Note: You can not insist on nitrogen at all temperatures.

The temperature at which the system switches from one gas to another depends upon whether the system is switching from helium to nitrogen or visa versa or, the temperature is going up or down.

If the gases are connected up incorrectly, the system is still in a position to protect itself. The controller contains only one gas detection chamber. This means that even if nitrogen is connected up to the

helium connector in the back of the controller, the system will detect the gas coming in and will behave appropriately.

4.5. HOLD and 'Un-HOLD'

A program can be paused at any time using the HOLD button; this will illuminate the HOLD lamp. To continue the program simply press the HOLD button at any time and the HOLD lamp will go out.

It is also possible to release the HOLD phase by pressing the START button.

4.6. Alarm conditions

The 700 Series N-HeliX Controller has a number of safety features. If there is an issue with the system, an alarm condition is indicated by an illuminated ALARM lamp and a warning will appear on the bottom screen of the controller. The following list describes all the warnings stored by the Controller.

Temp Warning – If the temperature error has reach 5K the controller will indicate a warning but will not shut down.

Pressure Warning – If a blockage forms inside the coldhead, the controller will sense the back pressure and trigger a pressure warning. To remove this ice blockage, restart the system and program a PURGE. A PURGE will heat up the system as quickly as possible.

Self-Check Fail – During the initialisation, the controller checks a variety of parameters to make sure that everything is connected properly and that there is continuity in all parts of the system. Try restarting the controller a number of times to see if the problem persists. If it does, contact Oxford Cryosystems.

Flow Rate Fail – As the controller is controlling the flow of gas through the system it will indicate if there is a gas flow problem. This could be due to a blockage or restriction, no source gas or an outward leak of source gas.

Temp Control Err – If the temperature error has reached 25K and the controller reads this value five times from the system, the controller will indicate a warning and shut down.

Gas Type Error – As the N-HeliX works with both helium and nitrogen, it has the ability to detect whether the gas is the correct type at any given temperature. For example, if a user tries to run with nitrogen at helium temperatures, the system will shut down with this error to avoid freezing the nitrogen.

Temp Reading Err – The controller received a nonsense reading from the temperature sensors.

Sensor Fail – If the controller received extreme values from the sensors, it will try to reset them. If the sensors fail to reset after five attempts, the controller will shut down with this error.

Brownout – If there is a brief interruption in the electrical supply to the controller, the controller will indicate a 'Brown Out' has occurred. The controller will continue to function normally.

Sink Overheat – If the controller overheats, there will be a Sink Overheat warning. This is often due to the covering of the fan on the underside of the controller.

PSU Overheat – If the controller overheats, there could be a PSU Overheat warning. This is often due to the covering of the fan on the underside of the controller.

Power Loss – When the power to a controller is cut, the controller will report a Power Loss error in the diagnostic screen when it is restarted.

Cryodrive Off? – The controller is connected to the Refrigerator Compressor (Cryodrive). If this connection is broken, the Cryodrive is switched off or there is no power to the Cryodrive, the controller will indicate it.

Cryodrive Error – There are a number of warnings given out by the Cryodrive. These include temperature warnings and pressure warnings. A temperature warning may arise due to the water temperature being too hot or too cold. A pressure warning may arise from a low helium pressure.

No Nitrogen – The N-HeliX actually detects the gases that are fed into the controller. If the wrong gas is fed into the nitrogen port of the system, it will indicate that there is no nitrogen. However, under these circumstances, it will not necessary shut down. The N-HeliX has no objection to using helium instead of nitrogen in the nitrogen port.

No Helium – The N-HeliX actually detects the gases that are fed into the controller. If the wrong gas (or not helium) is fed into the helium port of the system, it will indicate that there is no helium and shut down.

When there is a warning or if the system shuts down, the ALARM lamp will flash quickly and the buzzer will sound. See the diagnostics by pressing DISPLAY and report these to Oxford Cryosystems.

4.7. Safety features during power failures

The N-HeliX Controller is designed to protect itself and the sample during power interruptions.

It is possible for the Controller to maintain gas flow and not reset the Controller during electrical interruption indicated by the term 'Brown-Out' of between 0-2 seconds. If a 'Brown-Out' is detected, this is indicated on the screen.

4.8. Checklist to Start the N-HeliX running

Double check:

- ✓ Cryodrive has adequate water-cooling and switched on
- ✓ Helium Gas is connected to the back of the controller, set to 1 bar pressure and there is adequate supply remaining
- ✓ The Turbo Station is connected to the N-HeliX, switched on and the N-HeliX has a vacuum of $<10^{-5}$mbar
- ✓ The controller is switched on.

4.9. How to shut down the N-HeliX

To shut down the N-HeliX correctly, the user should program in an END phase into the Phase Table. END will ramp the N-HeliX to room temperature with no cryocooling, when it reaches this temperature, the N-HeliX will shut down. It is important to maintain the Helium gas flow during this procedure to prevent moisture from the air migrating up the nozzle.

In the case of an emergency, press the red STOP button.

Once the N-HeliX has shut down it is necessary to press the START button in the front of the controller if one wishes to continue using the system.

In the case of an unexpected shut down, press the START button, wait for the system to initialise and the record all the information on the Status Screen before switching the N-HeliX Controller off.

Note that not all these options will be available at any one time. The Controller will only allow those options that are possible.

4.9.1. N-HeliX shut downs

The N-HeliX Controller has been designed to fully protect the N-HeliX under its normal mode of operation as outlined in this manual. The control program will shut down the N-HeliX if:

1. The Gas Temp or Shield thermometer registers an error greater than ±25 K.
2. The Controller overheats.
3. A temperature sensor (Gas temperature or Shield temperature) fault occurs.
4. A Coldhead cable fault is detected.
5. The Cryodrive Shuts down unexpectedly (low charge pressure / lack of water cooling resulting in high temperature)

In each case the Status Screen variables are fixed and a suitable error message is displayed.

5. Measuring the True Crystal Temperature

The absolute measurement of temperature is generally very difficult, especially in the heterogeneous environment of an open cold stream. The flow rate in the N-HeliX has been designed to produce laminar flow for a few millimetres from the nozzle tip. Therefore, in order to have the crystal at a temperature close to that indicated on the Controller, we advise wherever possible to ensure that the crystal sample is 3.5 mm INTO the Beryllium shielding.

We do not recommend measuring the temperature with a thermocouple placed in the stream. In the heterogeneous environment of a narrow cold gas stream, there are several factors that lead to spurious voltages on the thermocouple, creating errors in apparent temperature of possibly tens of degrees! For instance, conduction of heat down the wires creates a heat leak. At the point of entry of the thermocouple wires into the stream a cold junction is formed whose temperature is much lower than the room temperature assumed by the Controller, thus making the measured temperature *appear* to be much higher than indicated. Also, the sharp temperature change at the interface between the cold stream and the surrounding warm air can induce stresses into the thermocouple wires and then generate spurious EMF.'s. We believe that the only satisfactory way to find the error in absolute temperature at the crystal position is to calibrate with a sample that undergoes a known phase transition or change of state. For instance, we have found that the low -temperature phase transition in the langbeinite $(NH_4)_2Cd_2(SO_4)_3$ was observed from intensity measurements to be in the range 88-89K (established elsewhere to be at 88 K). Similarly, lattice parameter measurements of sodium ammonium tartrate tetrahydrate gave a transition temperature in agreement to within 0.5K of the published value of 109 K. A most useful compilation of transitions in hundreds of crystals has been published by P. Tomaszewski (*Phase Transitions*, **38**, 127).

6. Running the N-HeliX with Cryopad

Cryopad is a PC program which allows remote monitoring and control of any 700 series Oxford Cryosystems device. This includes the 700 series Cryostream / Plus, Cobra / Plus, PheniX and N-HeliX systems.

6.1. Installing Cryopad

Install Cryopad from the CD also supplied with the Cryostream or download from the Oxford Cryosystems website, www.oxcryo.com/software/cryopad. If you experience problems with the web installer please install the Microsoft Visual C++ Redistributable Package as explained on the above web site.

6.2. Using Cryopad to run the N-HeliX

With the controller switched off connect a COM port from your PC to the port labelled SERIAL on the back of the controller. **This connection can be made with any standard M-F serial cable**. Now turn on the contoller and start Cryopad by selecting from the Oxford Cryosystems group in the Start Menu, or else by double-clicking the Cryopad logo on your desktop.

6.2.1. Connecting using the Settings page

The first time you use Cryopad you will need to select a COM port using the Settings page. If you know which COM port you are using then select it from the **Connect Using Port** menu. If you are using a non-standard COM port you may type its name directly here. Alternatively click the **Search…** button to display the Search dialog, which may be used at any time to scan your computer's COM ports for compatible devices.

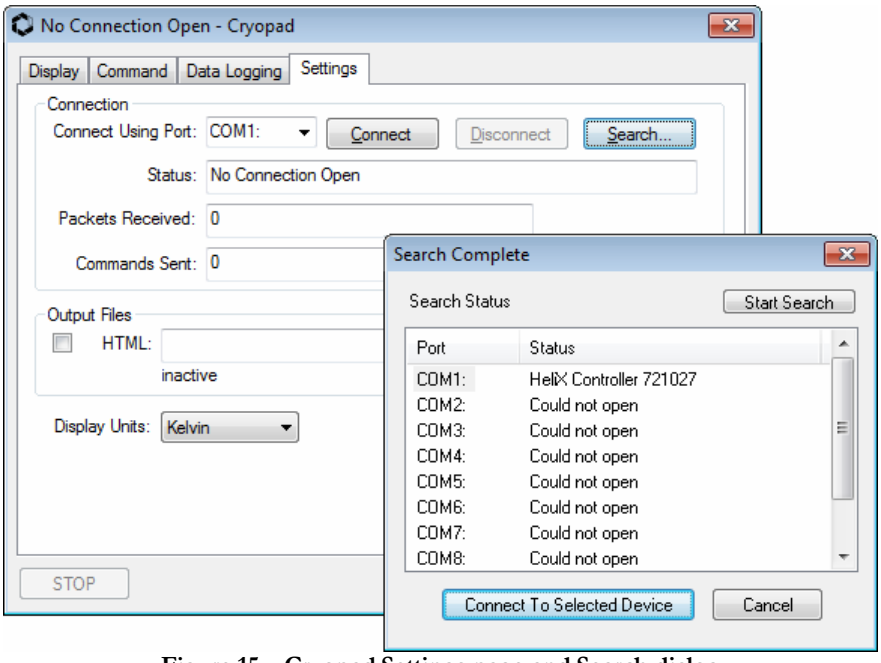


Figure 15 – Cryopad Settings page and Search dialog

The next time you run Cryopad your chosen COM port will be opened automatically, and a connection will be established as soon as a compatible device is detected. Should you need to change the COM port, switch to the Settings page, press Disconnect and repeat the above procedure. Should you wish to connect more than one device, run a new copy of Cryopad for each device and connect each one as described above.

6.2.2. The Display page

Once a connection has been established the Display page shows the live status of the device. The table below indicates the meaning of the various quantities displayed. After half an hour or so when the device has reached its normal operating values all the indicators will appear green.

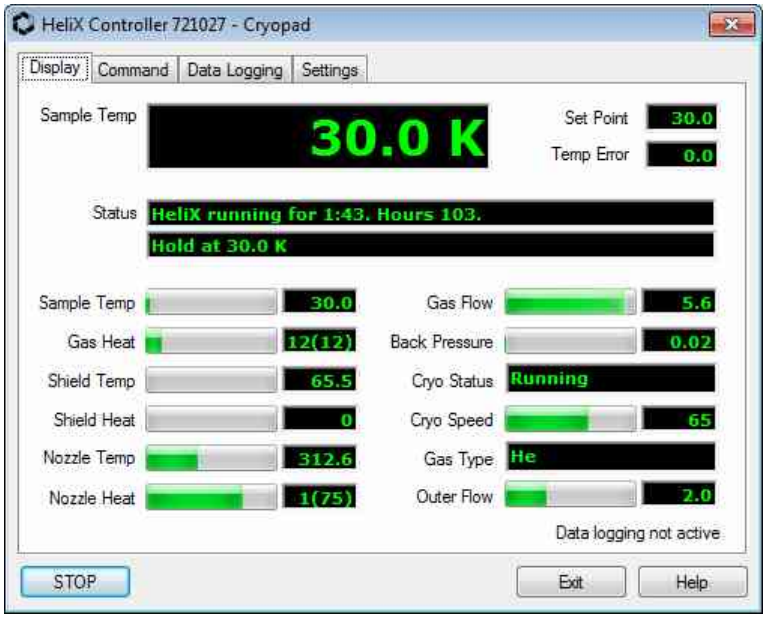


Figure 16 – Cryopad Display page

Data	Explanation
Sample Temp	The gas temperature at the crystal position
Set Point	The temperature to which N-HeliX is controlling
Temp Error	The difference between the Sample Temp and the Set Temp, except in a Cool phase, in which Temp Error is zero.
Status	Two lines of information indicating the current status of N-HeliX. Any errors or warnings raised by N-HeliX will be displayed here.
Gas Heat	The power to the sample heater, expressed as a percentage of full power.
Shield Temp	The temperature measured by the shield sensor.
Shield Heat	The power to the shield heater, expressed as a percentage of full power.
Nozzle Temp	The temperature measured by the nozzle sensor.
Nozzle Heat	The power to the nozzle heater, expressed as a percentage of full power.
Gas Flow	The gas flow in litres / minute.
Back Pressure	The back pressure in the gas line, measured in bar.
Cryo Status	An indication of the status of the Cryodrive.
Cryo Speed	The current Cryodrive speed, presented in RPM.
Gas Type	The current gas type. Possible values are N2, He or None. Whilst the gas type is changing this item may well read N2/He or He/N2.
Outer Flow	The flow of the shroud gas in litres / minute.

6.2.3. The Command page

The Command page allows commands to be sent to your N-HeliX exactly as if they were entered using the controller. Commands sent in this way will immediately overwrite the contents of the controller's Phase table. Refer to section 4 above for details of the commands.

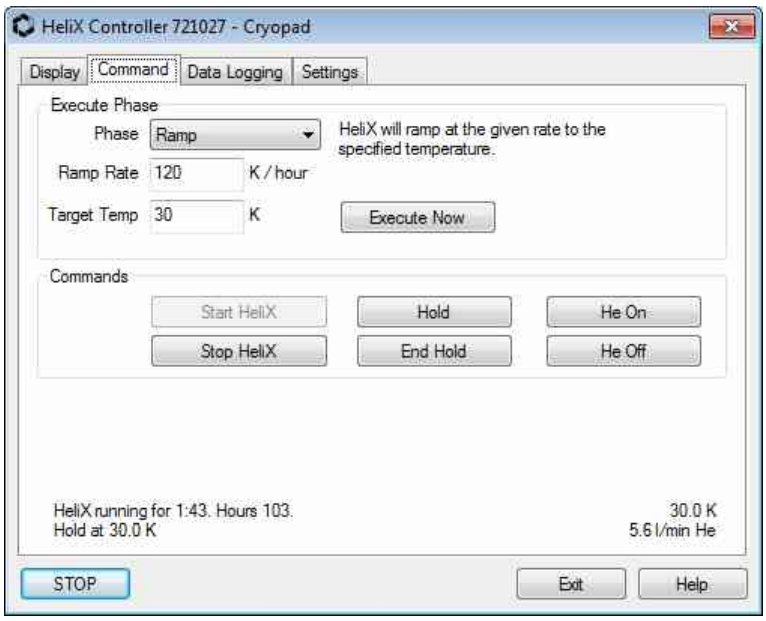


Figure 17 – Cryopad Command page

6.2.4. The Data Logging page

Cryopad allows data to be logged to a tab-delimited text file suitable for use in Excel or similar programs. Choose a file to which the data will be logged using the Log File item, and select the quantities of interest using the check boxes. The Interval item allows you to select the interval in seconds at which the data are logged. For monitoring purposes a 60 s interval is suitable whereas for diagnosing problems an interval of 1 s provides the most information but will produce a larger log file. Check the Logging Active box to commence logging.

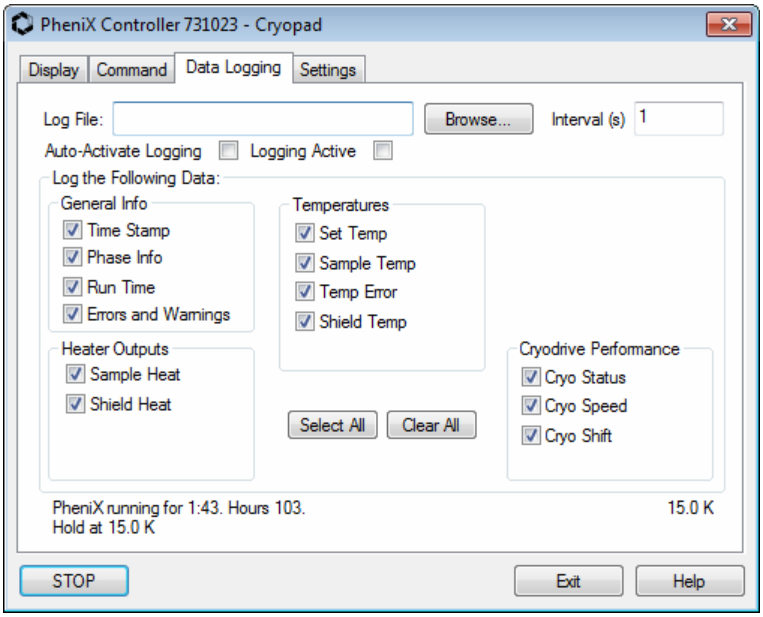


Figure 18 – Cryopad Data Logging page

7. Troubleshooting Guide

During the operation of the N-HeliX it will be necessary to routinely check many system parameters, such as Vacuum within the Coldhead, Gas flow, Cryodrive compressor pressure, Cryodrive water-cooling, and quantity of helium remaining. Good system maintenance and monitoring will help to avoid unexpected problems.

The following table lists possible symptoms and their remedy.

Observation	**Cause**	**Solution**
Unable to achieve base temperature of 28 K	Poor vacuum	Check vacuum system
	Low Cryodrive pressure / Cryodrive not functioning correctly	Check or service the Coolstar refrigerator and/or Cryodrive compressor
N-HeliX coldhead cold or wet through condensation	Loss of vacuum	Shut down N-HeliX **DO NOT USE!** Check vacuum system
Nozzle iced up	Nozzle heater broken	Check nozzle is warm to the touch.
	Shield gas flow too low / high	Check shield gas flow rate is at the optimum value. Check helium supply cylinder is delivering 1 bar outlet
Low rate of gas flow	Blockage due to poor quality helium gas	Allow N-HeliX to warm up. Check purity of helium gas or fit filter into supply line.
	Contamination due to Inwards leak	Check the gas tubing is fitted correctly into the quick-fit connectors

If the suggested solutions do not work, or another symptom presents, please contact Oxford Cryosystems (support@oxcryo.com) for free technical support.

8. N-HeliX User Maintenance

The N-HeliX has been designed to be as easy to use as possible and should run without the need for constant attention, once the user is accustomed to the system. However, some parts of the system will eventually require maintenance, with the following maximum service intervals:

Procedure	Maximum Service Interval
Oil Change For Vacuum Pump[1]	8,000 Hours
Maintenance of Helium level in Cryodrive 3.0[2]	As Required
N-HeliX Coldhead Swap-Out[3]	15,000 Hours
Replacement of Adsorber in Cryodrive 3.0[4]	15,000 Hours

Table 3 - N-HeliX Service Intervals

1. See Section 8.3 for details.

2. The Helium pressure in the Cryodrive should be topped up to 16.5 bar (240 psi) at rest. See Section 8.1 for details.

3. See Section 8.4 for details.

4. See Section 8.2 for details.

8.1. Topping up the Cryodrive with helium

The N-HeliX system consists of a closed helium gas circuit. The helium in this circuit will eventually leak out over time and will require topping up from a high pressure helium gas cylinder (99.9995%). **Note: that this should only be performed when the system is off.**

Re-charge the Cryodrive with helium if the helium pressure falls to below 15.5 bar.

CAUTION

Ensure that the interconnecting pipe work is capable of safely withstanding the maximum regulator delivery pressure.

WARNING

When you vent helium from the charge and vent adapter during the purging procedure, ensure that the vented gas is directed safely away from personnel.

CAUTION

You must re-charge the Cryodrive with 99.9995% helium. If you do not, you will contaminate the Cryodrive with impurities that will reduce its efficiency.

Re-charge the Cryodrive with helium if the helium pressure falls to below 15.5 bar. If you need to re-charge the Cryodrive frequently (for example, every 6 months or more often), there is probably a leak in your installation. Use a helium leak detector or other suitable method to find the leaks, and contact Oxford Cryosystems with the results, or for further guidance.

To recharge the system with Helium, please follow the instructions in Section 5.3 of the Cryodrive manual, supplied with the Cryodrive.

Note that if the helium pressure has fallen to 0 bar (atmospheric pressure), the helium should not be topped up. Instead, contact Oxford Cryosystems for advice on how to proceed.

8.2. Replacing the Cryodrive Adsorber

When the Cryodrive has been operating for approximately 16000 hours, you must replace the Adsorber with a new one to avoid permanent damage to the N-HeliX Coldhead. Please contact Oxford Cryosystems for more information.

The new Adsorber is supplied pressurised with helium, so you should not have to re-charge the Cryodrive with helium after you fit the new Adsorber. A de-pressurisation adapter is supplied with the new Adsorber.

If necessary, refer to the 'Cryodrive Operation & Instruction Guide' for details of how to connect and disconnect the self-sealing Aeroquip coupling used for the Adsorber in the Cryodrive.

WARNING

Do not bend over the internal pipe work when you fit and remove the Adsorber. After removal, the old Adsorber must be safely depressurised before disposal. The replacement Adsorber will be charged with helium to 16.5 bar. Always vent gas safely, directed away from personnel.

Replacing the Adsorber:

1. Switch the Cryodrive ON/OFF switch to OFF. Isolate the Cryodrive from the electrical supply.
2. Remove the lid of the Cryodrive.
3. Disconnect the helium supply hose located at the rear of the Cryodrive.
4. Disconnect the Aeroquip coupling from the Adsorber inlet.
5. Unscrew and remove the Adsorber rear panel locking nut and washer. Remove the single screw retaining the Adsorber inlet connection clamp plate. Lift the Adsorber out of its locating hole and remove the Adsorber. Remove the locking nut and clamp plate from the Adsorber inlet connection. Retain the clamp plate and screw, locking nut, washer and star washer.
6. Depressurise the old Adsorber by connecting the depressurisation adaptor to the Adsorber helium inlet and outlet coupling and tightening slowly by hand.
7. Remove the dust from the inlet and discharge self-sealing couplings of the new Adsorber. Fit the Adsorber clamp plate and locking nut to the Adsorber Aeroquip inlet connection.

8. Install the new Adsorber in position in the compressor unit and ensure that the locating pin is correctly engaged. Secure the new Adsorber in place using the nut and washers, and Adsorber clamp plate screw retained in step 5.
9. Re-connect the helium supply hose. Re-connect the internal Aeroquip on the Adsorber inlet.
10. Re-fit the lid of the Cryodrive.

Check that the pressure gauge reads 16.5±1.0 bar (239.31±14.5 psig). If the gauge reads below 15.5 bar (210 psig), add helium gas following the procedure detailed in Section 8.1.

►Record the date the new Adsorber is fitted. Also record the hours run from the N-HeliX controller hour counter.

8.3. Servicing the Turbomolecular Vacuum Pumping Station

The maintenance intervals for the Pfeiffer Turbomolecular Vacuum Pump should be carried out approximately every four year independent of operating hours.

Parts to be serviced:

- Change of operating fluid reservoir
- Change of turbopump bearing

Please contact Oxford Cryosystems for further assistance and purchase of these items. Alternatively you may wish to consult the Operating Manual supplied by Pfeiffer Vacuum Company.

8.4. Coldhead swap-out

The coldhead located inside the N-HeliX will eventually require a service due to the delicate seals and 'O' rings wearing out. The easiest and most efficient way to do this is to contact Oxford Cryosystems for a replacement coldhead swap-out. This will be sent to you and we then ask you to send us the old coldhead back.

9. Liquid and gaseous nitrogen safety sheet

9.1. General

These safety points are a guideline to outline the potential hazards and procedures involved in the handling of liquid or gaseous nitrogen. Anyone handling liquid or gaseous nitrogen should first inform their departmental or laboratory safety advisor and receive advice about local safety procedures.

All users are requested to read this safety sheet before handling the N-HeliX. Oxford Cryosystems accept no responsibility for injury or damage caused by the mishandling of liquid or gaseous nitrogen.

9.1.1. General properties

- Gaseous nitrogen is colourless, odourless and tasteless and is slightly lighter than air at equal temperatures; cold nitrogen vapour is, however, denser than atmospheric air.
- Liquid nitrogen is odourless, colourless and boils at -195.8°C. One volume of liquid nitrogen gives approximately 700 volumes of gas at ambient conditions.
- Nitrogen is not flammable. It is chemically inert, except at high temperatures and pressures. Its volume concentration in air is 78%.
- Liquid and cold gaseous nitrogen can cause severe burns or frostbite when in contact with the skin or respiratory tract.
- Gaseous and liquid nitrogen is non-corrosive.
- Nitrogen does not support life and acts as an asphyxiant.
- Nitrogen is intrinsically non-toxic.

9.2. Fire and explosion hazards

Gaseous and liquid nitrogen are non-flammable and do not, themselves, constitute a fire or explosion risk. However, both gaseous and liquid nitrogen are normally stored under pressure and the storage vessels whether gas cylinders or liquid tanks, should not be located in areas where there is a high risk of fire or where they may normally be exposed to excessive heat.

9.3. Health hazards

9.3.1. Asphyxia

Nitrogen, although non-toxic, can constitute an asphyxiation hazard through the displacement of the oxygen in the atmosphere. Nitrogen gas or oxygen depletion is not detectable by the normal human senses.

Oxygen is necessary to support life and its volume concentration is the atmosphere is 21%. At normal atmospheric pressure persons may be exposed to oxygen concentrations of 18% or even less, without adverse effects. However, the response of individuals to oxygen deprivation varies appreciably. The minimum oxygen content of breathing atmospheres should be 18% by volume but to ensure a wider margin of operational safety it is recommended that persons are not exposed to atmospheres in which the oxygen concentration is, or may become, less than 20% by volume.

Symptoms of oxygen deprivation, such as increased pulse and rate of breathing, fatigue, and abnormal perceptions or responses, may be apparent at an oxygen concentration of 16%.

Permanent brain damage or death may arise from breathing atmospheres containing less than 10% oxygen. Initial symptoms will include nausea, vomiting and gasping respiration. Persons exposed to such atmospheres may be unable to help themselves or warn others of their predicament. The symptoms are an inadequate warning of the hazard.

WARNING

Breathing a pure nitrogen atmosphere will produce immediate loss of consciousness and almost immediate death.

9.3.2. Cold burns

Liquid and cold nitrogen vapours or gases can produce effects on the skin similar to a burn. Naked parts of the body coming into contact with un-insulated parts of equipment may also stick fast (as all available moisture is frozen) and the flesh may be torn on removal.

9.3.3. Frostbite

Severe or prolonged exposure to cold nitrogen vapour or gases can cause frostbite. Local pain usually gives warning of freezing but sometimes no pain is experienced. Frozen tissues are painless and appear waxy with a pallid yellowish colour. Thawing of the frozen tissues can cause intense pain. Shock may also occur if the burns are at all extensive.

9.3.4. Effect of cold on Lungs

Prolonged breathing of extremely cold atmospheres may damage the lungs.

9.3.5. Hypothermia

Low environmental temperatures can cause hypothermia and all persons at risk should wear warm clothing. Hypothermia is possible in any environmental temperature below 10ºC but susceptibility depends on time, temperature and the individual. Older persons are more likely to be affected. Individuals suffering from hypothermia may find that their physical and mental reactions are adversely affected.

9.4. Precautions

9.4.1. Operations and maintenance

It is essential that operations involving the use of gaseous or liquid nitrogen, particularly where large quantities are used, are conducted in well-ventilated areas to prevent the formation of oxygen deficient atmospheres.

Ideally, nitrogen should be vented into the open air well away from areas frequented by personnel. It should never be released or vented into enclosed areas or buildings where the ventilation is inadequate. Cold nitrogen vapours are denser than air and can accumulate in low lying areas such as pits and trenches.

Where large spills of liquid nitrogen occur a fog forms in the vicinity of the spill caused by the condensation of water vapour in the surrounding air. The fog, in addition to severely reducing visibility, may contain oxygen concentrations appreciably lower than that of the air presenting a local asphyxiation hazard.

9.4.2. Personnel protection

Persons handling equipment in service with liquid nitrogen should wear protective face shields, loose fitting gauntlets and safety footwear.

9.4.3. Emergencies

In the event of an accident or emergency the instructions below should be implemented without delay.

9.4.4. Asphyxiation

Persons showing symptoms of oxygen deprivation should be moved immediately to a normal atmosphere. Persons who are unconscious or not breathing must receive immediate first aid. Medical assistance should be summoned without delay. First aid measures included inspection of the victim's airway for obstruction, artificial respiration and simultaneous administration of oxygen. **These procedures should only be carried out by a trained first aid staff.** The injured should be kept warm and resting.

It is important that the personnel carrying out rescue operations should minimise the risk to themselves.

9.4.5. Treatment of cold burns and frostbite

Cold burns should receive medical attention as quickly as possible. However, such injuries are not an everyday occurrence and doctors, hospital staff or works first aid personnel may not be aware of the basic methods of treatment. The following notes describe the first aid treatment and recommended advice for further treatment to be given by a medical practitioner or a hospital.

9.5. First aid

In severe cases summon medical attention immediately. Flush affected areas of skin with copious quantities of tepid water to reduce freezing of tissue. Loosen any clothing that may restrict blood circulation. Move the victim to a warm place but not to a hot environment and do not apply direct heat to the affected parts. Every effort should be made to protect frozen parts from infection and further injury. Dry, sterilised bulky dressings may be used but should not be applied so tightly that blood circulation is restricted.

9.5.1. Treatment by medical practitioner or hospital

1. Remove any clothing that may constrict the circulation to the frozen area. Remove patient to sick bay or hospital.

2. Immediately place the part of the body exposed to the cryogenic material in a water bath which has a temperature of not less than 40°C but no more than 45°C. **Never use dry heat or hot water.** Temperatures in excess of 45°C will superimpose a burn upon the frozen tissue.

3. If there has been a massive exposure to the super cooled material so that the general body temperature is depressed, the patient must be re-warmed gradually. Shock may occur during re-warming, especially if this is rapid.

4. Frozen tissues are painless and appear waxy with a pallid yellowish colour. They become painful, swollen and very prone to infection when thawed. Therefore, do not re-warm rapidly if the accident occurs in the field and the patient cannot be transported to hospital immediately. Thawing may take from 15-60 minutes and should be continued until the blue, pale colour of the skin turns to pink or red. Morphine, or some potent analgesic, is required to control the pain during thawing and should be administered under professional medical supervision.

5. If the frozen part of the body has thawed by the time medical attention has been obtained, do not re-warm. Under these circumstances cover the area with dry sterile dressings and a large bulky protective covering.

6. Administer a tetanus booster after hospitalisation.

9.5.2. Hypothermia

Persons suspected to be suffering from hypothermia should be wrapped in blankets and moved to a warm place. Slow restoration of temperature is necessary and forms of locally applied heat should not be used. Summon medical attention.

9.5.3. Liquid nitrogen spillage

If large spills of liquid nitrogen occur, large quantities of water should be used to increase the rate of liquid vaporisation.

10. Technical Support

To allow Oxford Cryosystems to offer fast and accurate technical support, please quote your N-HeliX Serial Number with all technical issues. It is worth keeping a record of this number in a convenient place:

N-HeliX SERIAL NUMBER
This N-HeliX serial number is _____

Before you return your equipment you must warn Oxford Cryosystems by contacting us.

Oxford Cryosystems Ltd contact details:

Email: support@oxcryo.com

Phone: +44 (0)1993 883488

Fax: +44 (0)1993 883988

10.1. Returns procedure

Use the following procedure to return ANY items for repair.

1. Contact Oxford Cryosystems and obtain an 'RMA' number for your equipment which must be written on each box that you return. Without this number we may reject packages. You will also be emailed a form which you must fill in and email or fax back to us prior to sending your package(s).

2. Remove all traces of dangerous substances and any accessories that will be returned to Oxford Cryosystems. Drain all fluids and lubricants from the equipment and its accessories.

3. Disconnect all accessories from the equipment. Safely dispose of the filter elements from any oil mist filters.

4. Seal up all of the equipment's inlets and outlets (including those where accessories were attached). You may seal the inlets and outlets with blanking flanges or heavy gauge PVC tape.

5. Seal contaminated equipment in a thick polythene bag. If you do not have a polythene bag large enough to contain the equipment, you can use a thick polythene sheet.

6. If the equipment is large, strap the equipment and its accessories to a wooden pallet. Preferably, the pallet should be no larger than 510 mm x 915 mm (20"x 35"); contact Oxford Cryosystems if you cannot meet this requirement.

7. If the equipment is too small to be strapped to a pallet, pack it in a suitable strong box.

8. If the equipment is contaminated, label the pallet (or box) in accordance with laws covering the transport of dangerous substances.

Oxford Cryosystems - Warranty Certificate

This warranty is subject to the Oxford Cryosystems Ltd's (OCL) Terms and Conditions of Sale.

OCL warrants to the Buyer that the goods sold for use hereunder will be free from defects in material and workmanship under normal use and operation for 12 months from the date of shipment from OCL's premises.

In order to obtain the benefits of the warranty the Buyer must first notify OCL of the defects. An OCL representative will verify the nature of the defect and if it is covered by this warranty, OCL will issue the Buyer with a RMA number and provide the Buyer with instructions on how to return the goods to OCL. The Buyer must return the goods according to instructions from OCL, complete with a written description of the claimed defect and RMA number. The goods should be packed safely, preferably in its original packaging prior to return.

The Buyer shall meet the cost of shipping the defective goods to OCL and OCL will pay any return costs to the Buyer

OCL's obligation under this warranty is limited to its option to repair or replace goods that are proven to be defective when used under normal operating conditions and within specification.. This warranty does not cover any changes made by the customer, depreciation of the goods or claims for compensation.

No warranty is given for damage resulting from misuse or fair wear and tear. In addition, this warranty does not cover any costs incurred in damage arising from the dismantling or reassembly of any of the goods, or for consequential losses of time or materials caused by Cryostream failure.

Registration
In order for us to be able to provide fast and effective service, you should register your system with us. Please send the serial number of the system (found engraved on the Coldhead) to support@oxcryo.com, together with your full contact details.

To make contact with Oxford Cryosystems you can telephone, fax, or email us at:

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